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(54) **Improvements in granulation**

(57) A granulation process comprises mixing pulverulent wax or polymer with pulverulent additive free from starch and feeding the mixture through a die of a moulding machine at a temperature such that in the die the additive is pressed into the softened wax or polymer.

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SPECIFICATION

## Improvements in granulation

The present invention concerns improvements in granulation.

The incorporation of additives, for example pigments, into polymers before processing for example by extrusion and fabrication is normal practice in the plastics and rubber processing industries. Additive in powder form tends to separate from a mixture with polymer in granule form or "hold-up" in the hopper of an extruder causing problems in achieving uniform distribution. Since the polymers are most often in the form of granules it is therefore usually necessary to convert the powdered additives into granules so that they can be efficiently handled by plastics processing plant, for example, an extruder, a machine incorporating an extruder or a fluxing mill with two rollers. This is usually achieved by masterbatching which is an expensive process necessitating high capital investment for relatively small throughput.

There is therefore a need for a granulation process using less expensive equipment which is capable of larger throughputs.

In pelleting animal feed rations containing starch granules some of the granules are gelatinised in the extrusion die and this serves to hold the pellets together.

We have found that pulverulent waxes or polymers can perform a similar role to that of gelatinised starch in binding animal feed pellets and enable additives to be produced in pellet form.

According to the present invention therefore, a process is provided which comprises mixing pulverulent wax or polymer with pulverulent additive free from starch and feeding the mixture through a die of a moulding machine at a temperature such that in the die the additive is pressed into the softened wax or polymer.

The polymer may be, for example, a synthetic resin having carbon-to-carbon linkages e.g. polyethylene or polypropylene. The wax may be natural or synthetic.

The wax or polymer, whichever is used, must of course be compatible with the polymer to which the pellets are eventually added.

The additive may be a single compound such as a pulverulent pigment or a mixture of compounds.

The moulding machine may be, for example, a pellet machine as used in the animal feed industry and described in Chapter 13 of "Compound Milling" by N.O. Simmons (published by Leonard Hill Ltd., London).

By means of the process of this invention pellets of acceptable durability can be prepared, that is, they are sufficiently durable to withstand mechanical handling, transport and storage in containers without breaking down to form an amount of dust which when mixed with polymer granules would cause processing problems.

The invention is illustrated by the following Examples.

*Example 1.*

Titanium dioxide (RFC 5 by B.T.P. Tioxide Ltd.) and powdered low density polyethylene (019-040 "Alkathene", I.C.I. Ltd.) Melt Flow Index 20, were dry blended in the ratio of equal parts by weight. The mixture was heated to 115°C and then continuously transferred to a Christy and Norris Pellet Press; the die used had 0.32 cm diameter holes of depth 2.5 cm. Well formed pellets of acceptable durability were produced. These pellets when mixed with low density polyethylene granules of density 0.916 and Melt Flow Index 1 in the ratio of 1 part by weight of pellets to 24 parts by weight of polyethylene, were fed to the hopper of a conventional film-blowing extrusion machine (a 60 mm screw diameter with L:D ratio of 31:1 made by Afex A.G. of Switzerland) and converted to film of 60 micrometre thickness. The titanium dioxide was seen to be uniformly dispersed and was present at a concentration of 2% by weight.

*Example 2.*

Beige pigment (DC8720 by Blythe Colours Ltd. Stoke-on-Trent) and powdered paraffin wax M.Pt. 57-60°C (Type 135/40 Astor Chemical Ltd. West Drayton) were dry blended in the ratio of equal parts by weight. The mixture was then cooled to −10°C and continuously transferred to a Christy and Norris Pellet Press fitted with the same die as in Example 1. Well formed pellets of acceptable durability were produced. A mixture of these pellets with polyethylene granules in such proportion as to provide a pigment concentration of 2% by weight in the final product was satisfactorily converted into film as in Example 1.

*Example 3.*

Calcium oxide (Sturge Chemicals Ltd.), oil (isooctyloleate by Unichem Chemicals Ltd., Bebington) and powdered paraffin wax Type 135/40 (melting point range 57-60°C) were blended in the ratio of 57 parts by weight of calcium oxide, 3 parts by weight of oil and 14 parts by weight of paraffin wax. This mixture was then continuously transferred at room temperature to a Christy and Norris Pellet Press fitted with the same die as in Example 1. Well formed pellets of acceptable durability were produced. A mixture of these pellets with polyethylene granules in such proportion as to provide a calcium oxide concentration of 3.1% by weight was satisfactorily converted to film as in Examples 1 and 2.

*Example 4.*

Pellets containing titanium dioxide and low density polyethylene prepared as in Example 1, were mixed with polyethylene granules (Alkathene, I.C.I. Ltd, WSM 78) Melt Flow Index 36 and density 0.919 in the proportions of 4% by weight of pellets and 96% by weight of granules. This mixture was fed to the hopper of an injection moulding machine (Butler Smith 100/60) fitted with a tensile bar and disc moulding tool and converted to a satisfactory injection moulding. The titanium dioxide was seen to be uniformly dispersed in the moulding and was present in a concentration of 2% by weight.

*Example 5.*

Titanium dioxide and powdered paraffin wax as in Examples 1 and 2, were blended with oil (soya bean oil) in the proportions of 50% by weight of titanium dioxide, 40% by weight paraffin wax and 10% by weight oil. The mixture was then fed at room temperature to a Christy and Norris Farm Type Pellet Press as in Example 1. Well formed pellets of acceptable durability were produced. A mixture of these pellets with polyethylene granules (as in Example 4) in the proportions of 4% by weight pellets to 96% granules was converted to a satisfactory injection moulding as in Example 4. The titanium dioxide was seen to be uniformly dispersed in the moulding and was present at a concentration of 2% by weight.

*Example 6.*

Mercapto Benzthiozole Disulphide (Vanax MBTS by R.T. Vanderbilt Co. Inc.) Powdered Paraffin Wax (as in Example 2), Calcium Carbonate (Snowcal 8/SW by C.M.C.Ltd U.K.) and Stearic Acid (Acidchem Ltd. Malaysia), were dry blended in the ratio by weight of 75 parts : 9 parts : 5 parts : 11 parts respectively. The mixture was heated to 53°C and then continuously transferred to a Christy and Norris Pellet Press fitted with the same die as in Example 1. Well-formed pellets of acceptable durability were produced. The pellets were tested, using a Haake Rheomix type 600 (supplied by M.S.E. Ltd. U.K.), in the following formulation in comparison with MBTS powder:-

| | | |
|---|---|---|
| Styrene Butadiene Rubber | Incarb 5609 by the International Synthetic Rubber Co.Ltd., U.K. | 100 parts |
| Reinforcing Carbon Black | | 90 parts |
| Aromatic Process Oil | | 70 parts |
| Stearic Acid | | 1 part |
| Zinc Oxide | Barking Zinc Oxide Ltd. | 5 parts |
| Sulphur | Seaton Chemical Dev. Ltd., U.K. | 2 parts |
| MBTS Samples * | | * |

| *MBTS Powder* | *MBTS Pellet* |
|---|---|
| 1.0 parts | 1.33 parts |
| 1.25 parts | 1.67 parts |
| 1.5 parts | 2.0 parts |

The Haake rheomix was run at 120°C and 45 r.p.m. and the viscosity and scorch times plotted. No measurable differences were noted on viscosity or scorch times for comparable quantities of powder or pellets.

*Example 7*

N(Cyclohexylthio) phthalimide (Santogard PVI by Monsanto Ltd.), powdered Paraffin Wax (as in Example 2), Calcium Carbonate (as in Example 6) and Mineral Oil (Paraffinic Mineral Oil Gulfpar — Gulf U.K.) were dry-blended in the ratio by weight of 50 parts : 8.5 parts : 40 parts : 1.5 parts respectively. The mixture was heated to 53°C and then continuously transferred to a Christy and Norris Pellet Press fitted with the same die as in Example 1. Well-formed pellets of acceptable durability were produced. These pellets were tested with the Haake Rheomix in the following formulation and compared with PVI powder:-

| | | |
|---|---|---|
| Styrene Butadiene Rubber | Incarb 5609 by the International Synthetic Rubber Co.Ltd., U.K. | 100 parts |
| Reinforcing Carbon Black | | 90 parts |
| Aromatic Process Oil | | 70 parts |
| Stearic Acid | | 1 part |
| Zinc Oxide | Barking Zinc Oxide Ltd. | 5 parts |
| Sulphur | Seaton Chemical Dev.Ltd., U.K. | 2 parts |
| CBS/PVI composition* | | * |

(CBS: N-cyclohexyl-2-benzo-thiozole sulphenamide VANAXCBS by R.T. Vanderbilt Co.Inc.)

| *CONTROL* *CBS Powder/PVI Powder* | *TEST* *CBS Powder/PVI Pellet* |
|---|---|
| 1.0/0.1 | 1.0/0.2 |
| 1.25/0.1 | 1.25/0.2 |
| 1.5/0.1 | 1.5/0.2 |

The Haake rheomix was run at 120°C and 45 r.p.m. and the viscosity and scorch times plotted. No measurable differences were noted on viscosity or scorch times for comparable quantities of powder or pellets.

*Example 8*

Zinc Oxide (as in Example 6), powdered Paraffin Wax and Soya Bean Oil were dry blended in the ratio by weight of 85 Parts : 10 parts : 5 parts respectively. The mixture was heated to 55°C and then continuously transferred to a Christy and Norris Pellet Press fitted with the same die as in Example 1. Well formed pellets of acceptable durability were produced.

These pellets were tested in the Haake rheomix run at 70°C and charged with 67g of soft Butyl Rubber (a commercially available innertube compound of 40 Shore hardness) and 5g of pellets for a total of approximately 20 minutes. The rubber stock was rolled into a ball and cut with a sharp knife into four segments and each cut surface was examined with × 4 magnifier at 3 cms focal length (Textile thread count magnifier used with field of view 1.8cms square). No undispersed particles were visible.

CLAIMS

1. A granulation process which comprises mixing pulverulent wax or polymer with pulverulent additive free from starch and feeding the mixture through a die of a moulding machine at a temperature such that in the die the additive is pressed into the softened wax or polymer.

2. A granulation process as claimed in claim 1 in which the polymer is a synthetic resin having carbon to carbon linkages.

3. A granulation process as claimed in claim 2, in which the synthetic resin is polyethylene or polypropylene.

4. A granulation process as claimed in any of claims 1 to 3, in which the additive is a pulverulent pigment.

5. A granulation process as claimed in claim 1, substantially as hereinbefore described in any one of the Examples.

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